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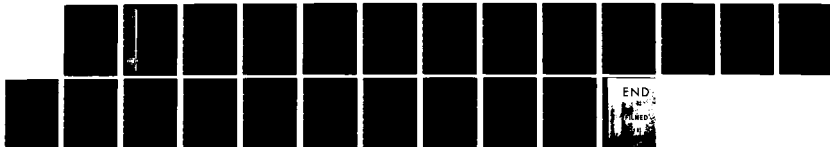
IDENTIFICATION AND CHARACTERIZATION OF ELASTOMER
VULCANIZATE MATERIALS SS. (U) ARMY MOBILITY EQUIPMENT
RESEARCH AND DEVELOPMENT COMMAND FORT. R G JAMISON
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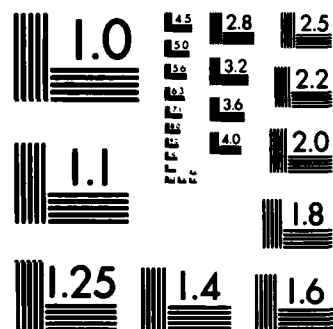
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IDENTIFICATION AND CHARACTERIZATION OF ELASTOMER
VULCANIZATE MATERIALS USING PYROLYTIC GAS LIQUID-
PHASE CHROMATOGRAPHY

by
Robert G. Jamison

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) <p>The objective of this program was to develop a capability differentiating between different types of elastomer vulcanizate currently used in military vehicles and equipment systems including those commonly found in tank hydraulic and gun control systems. The results of this study show that the proposed method can be used to determine the exact type of elastomer polymer and, thereby, can assure that the proper elastomer components are used in the system intended for use.</p>														

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CONTENTS

Section	Title	Page
	ILLUSTRATIONS	iv
I	INTRODUCTION	1
II	DETAILS OF TEST	3
III	RESULTS OF TEST	5
IV	DISCUSSION AND CONCLUSIONS	13



ILLUSTRATIONS

Figure	Title	Page
1	Predominant Peak Identifies from Pyrolytic Gas-Liquid Phase Chromatography Using a Flame	4
2	Synthetic Rubber "L"	7
3	Greentweed 964	8
4	Parkers N-756	9
5	Parkers N-304	10
6	Butyl "N" Rubber	11
7	Teflon	12

IDENTIFICATION AND CHARACTERIZATION OF ELASTOMER VULCANIZATE MATERIALS USING PYROLYTIC GAS LIQUID-PHASE CHROMATOGRAPHY

I. INTRODUCTION

All hydraulic systems of military equipment and commercial construction equipment contain elastomer components such as diaphragms, hoses, O-rings, etc., etc., that are in intimate contact with hydraulic fluid at various temperatures and pressures. In order for a system to operate effectively, the elastomer parts must not be adversely affected by the hydraulic fluid and must not degrade or contaminate other components in the system. The type of elastomer used to fabricate the various components is dictated by the type of hydraulic fluid in the system which, in turn, is determined by the type of hydraulic system and service conditions. In general, hydraulic fluids are classified as either "Petroleum Base Hydraulic Fluids" or "Non-Petroleum Base Hydraulic Fluids." Petroleum base hydraulic fluids include a wide variety of materials produced from different grades of crude oils and additives, and their use as hydraulic or power transmission fluids far surpasses all other classes. The non-petroleum base fluids include phosphate esters, silicate esters, organic acid esters, glycols, polysiloxanes, castor oils, poly alkylene glycols, oil-water emulsions, halocarbons, polyphenyl ethers, etc.

A large variety of vulcanized elastomer compounds of natural rubber, reclaimed rubber, and synthetic rubber, alone or in combination, are used for manufacturing products for a variety of system applications. These products include tires, belts, hose, mats, insulation for wire, shock absorbers, packings, and seals. The rubber may take the form of solid rubber or cellular (sponge) rubber. Since these components are used for a broad spectrum of service conditions and different environments, it is important to select elastomers possessing the necessary physical and chemical properties for a specific application. Consequently, a large number of different kinds of rubber are required to serve the many needs of the automotive industry. Some of the elastomers used are natural rubber (NR), styrene-butadiene rubber (SBR), acrylonitrile-butadiene rubber (NBR), butadiene rubber (BR), butyl rubber (IIR), chlorobutadiene or neoprene rubber (CR), chlorosulfonated polyethylene rubber (CSR), ethylene propylene rubber (EPM), fluorocarbon rubber (FR), isoprene or synthetic rubber (IR), polyacrylic rubber (AR), polysulfide (SR), polyurethane rubber (UR), silicone rubber (SR), and fluorosilicone rubber (FSR). This report is concerned, primarily, with the characterization of elastomeric components of hydraulic and gun recoil systems; however, the technique has application to other automotive elastomer materials.

A variety of synthetic and natural elastomers are used for the construction of hose and tubing for automotive applications. Hose may be composed of one class of elastomer throughout or of re-enforced laminates having one class of material in the inner tube and another material in the cover. The inner surface of the hose must not be degraded by the fluid and must not contaminate or deteriorate any part of the system. In addition, the outside of the hose must resist attack from oil, gasoline, grease, and water; and the hose must not crack or deform over the normal range of operating temperatures.

Pyrolytic gas liquid-phase chromatography (PGC) offers a convenient approach to the analysis of macromolecules without the necessity of chemical pretreatment. In PGC procedures, elastomers are thermally decomposed using a Curie-point apparatus, microoven, laser beam, or heated filament. Decomposition products are directed to a gas chromatograph (GC) where they are separated by standard GC technique. Generally, the thermal decomposition of polymers proceeds by three basic pyrolytic reactions. First, highly crosslinked polymers tend to carbonize. As a result, they produce pyrograms that are difficult to interpret and not easily referred back to the starting material. Second, some linear polymers will rupture between the side chain and polymer backbone producing fragments characteristic of the side chain. In most cases, pyrograms are characteristic of the polymer. The final and most useful type of pyrolysis occurs when the polymer backbone ruptures yielding monomers of the polymer. This type of decomposition is favored by quarternary substituted carbons on the polymer backbone. Yields of monomer, 90 percent or better, are generally obtained. Fortunately, most elastomer pyrolysis reactions generate products that yield pyrograms characteristic of the polymer from which they are derived, thereby permitting their identification in automotive rubber products.

The analysis of elastomers by classical methods of analysis is made possible only after extensive chemical treatment of the material, including the application of a battery of qualitative and quantitative tests. The ease and convenience of pyrolytic gas liquid-phase chromatography, however, has stimulated numerous investigations resulting in a large number of publications related to the analysis of natural and synthetic elastomers. Excellent comprehensive reviews of the literature have been presented by Audebert¹ and Stevens.² Some investigations were directed to a narrow range of blends and copolymer systems,^{3 4 5 6 7} whereas other studies were of much broader scope and included a large number of polymers, copolymers, and blends.^{8 9} Cole, et al.,⁹ investigated the pyrolysis characteristics of 46

¹ Audebert, R., *Ann. Chim.* 3, 49 (1968).

² Stevens, M. P., *Characterization and Analysis of Polymers by Gas Chromatography*, Marcel Dekker, New York (1969).

³ Umezawa, Y., Hasebe, Y., Hirai, N., and Furuse, T., *Nippon Gomu Kyokaishi*, 39, 497 (1966).

⁴ Perry, S. G., *J. Gas Chromatog.* 5, 77 (1967).

⁵ Mlejnek, O., *Chem. Průmysl*, 11, 604 (1961).

⁶ Cox, B. C. and Ellis, B., *Anal. Chem.* 36, 90 (1964).

⁷ Krishen, Anoop, *ibid.*, 44, 494 (1972).

⁸ Mashiko, Y., Echizen, A., and Nakao, A., *Kogyo Kagaku Zasshi*, 68, 1206 (1965).

⁹ Cole, H. M., Peterson, D. L., Slijaka, V. A., and Smith, D. S., *Rubber Chem. Technol.* 39, 259-277 (1966).

polymers using flame ionization and electron capture detection, temperature programming, and dual columns. Although the value of PGC as an analytical tool has been firmly established and documented in the literature, it suffers from one major problem; results are difficult to reproduce in different laboratories. A possible root cause of disagreement may be attributed to the diversity of pyrolysis apparatus. In fact, the history of PGC appears to be a continuing search for the ultimate pyrolysis unit that will provide reproducible results from one laboratory to another. Nevertheless, the technique is extremely useful when a reference library of pyrograms is produced within the laboratory using identical pyrolysis and GC operating conditions for standard and sample analyses.

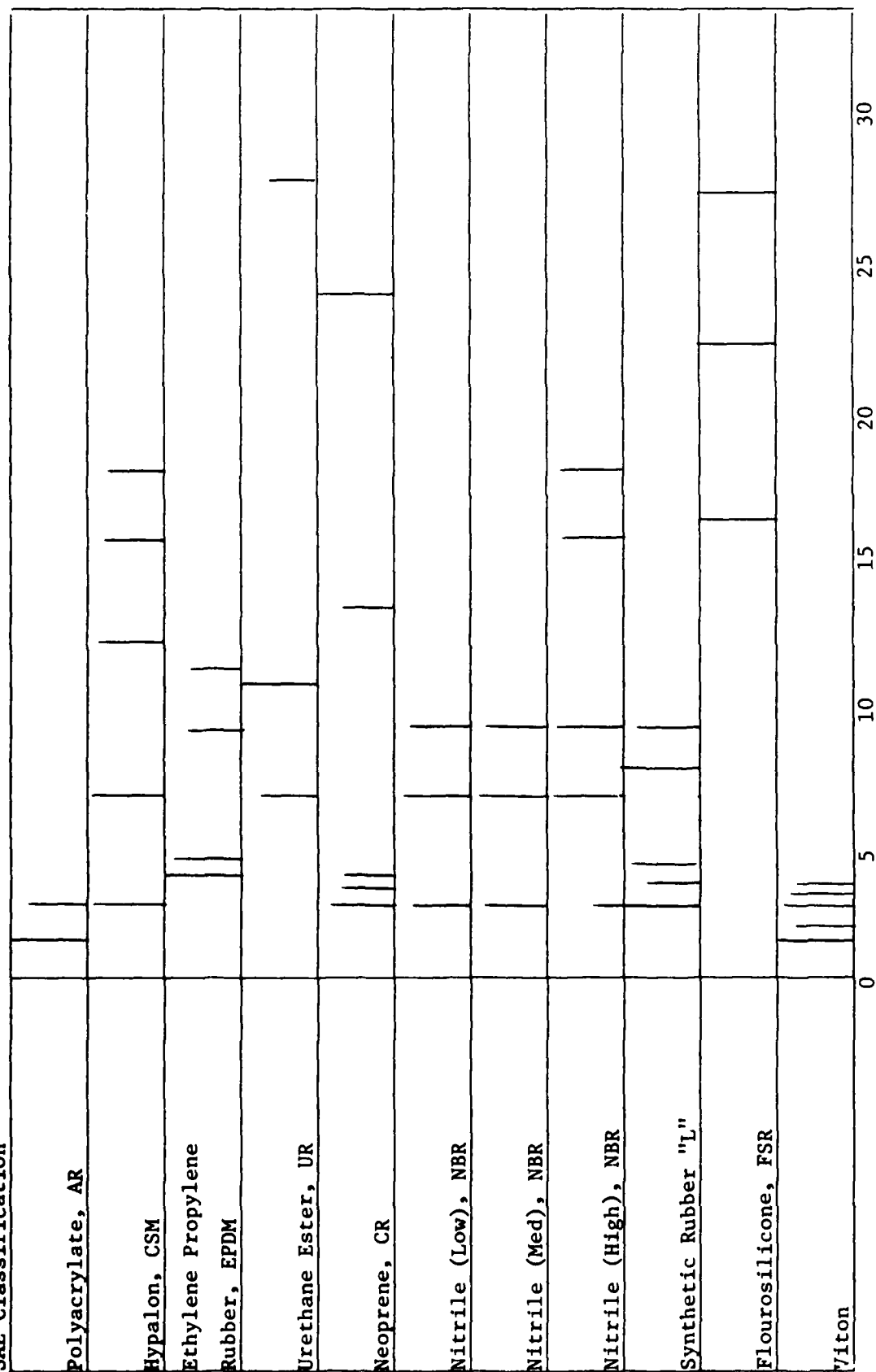
Several years ago, a report was published¹⁰ which described in detail the application of the combined technology (e.g., pyrolysis gas liquid-phase chromatography) in defining elastomer vulcanizate compositions. The objective in this report is to provide a follow-on investigation using an improved detection-capability with greater resolution. As in the previous study, the most commonly used elastomer types were analyzed by this improved technique, and practical applications revealing the utility of this methodology are demonstrated. The individual materials were pyrolyzed and their "fingerprint" pyrograms were developed. These, in turn, were then compared with pyrograms developed using reference elastomer vulcanizates of known compositions. To illustrate this point, Figure 1 identifies the predominant peak locations on reference pyrograms developed from several commonly-used elastomeric vulcanizates. Additional details on the procedures employed and examples of their applications will be presented.

II. DETAILS OF TEST

A Hewlett Packard Gas Chromatograph, Model 5834A, with a pyrolysis unit (Pyroprobe 18580A) was used for this test. The column used was an 18-ft by 1/8-in. stainless steel with 20 percent DC11 on Chromasorb W (60-80 mesh). To obtain good reproducibility and repeatability, it is critical that the sample size be consistently 0.0004 ± 0.00005 g.

¹⁰ Esposito, George and Jamison, Robert, Characterization of Elastomeric Materials Used in Automotive Brake and Cooling Systems by Pyrolytic Gas Chromatography, CCL Report No. 309, July 1972.

Elastomer Type and
SAE Classification



Retention Time, (Min)

Figure 1. Predominant peak identifications from pyrolytic gas-liquid phase chromatography using a flame.

The conditions for operation of the gas chromatograph are:

Initial Temperature: 50° C.

Rate: 6°/min.

Final Temperature: 250° C.

Injection Temperature: 250° C.

Flame Ionization Detector Temperature: 225° C.

Chart Speed: 10.

Attenuation: 10.

Helium Flow: 30 ml/min.

The pyroprobe conditions are as follows:

Interface Temperature: 225° C.

Interval: 10 s.

Final Temperature: 950° C.

Element Type: Quartz Tube with Coil Heaters.

A thin slice of elastomer sample weighing 0.4 ± 0.05 mg is placed in the quartz tube of the pyrolyzer probe which is inserted into the injection port of the chromatograph. The automated pyrolysis cycle and temperature-programming mechanism are engaged, and the chromatogram is developed using the operating conditions described above.

III. RESULTS OF TESTS

A series of nitrile elastomers which are copolymers of butadiene and acrylonitrile containing differing amounts of acrylonitrile were run in order to determine if the various grades could be identified. The identification was achieved by using the retention time (tr) of 2.96 min (± 5 percent) and measuring the peak height. It is evident that with increased amounts of acrylonitrile in the elastomer there is an increase in peak height (all except the 44 percent as shown in the table).

Known Nitrile Elastomers Versus Peak Height

Nitriles	Known	Peak Height in mm
209-10	20.4 %	32
N-216-1	22.0 %	54
N-216	24.25 %	56
N-206	44.5 %	10

A formulated standard nitrile elastomer compound known as Standard Rubber "L" is used in fluid testing and is specified in Mil-Specs MIL-H-5606,¹¹ MIL-H-6083,¹² MIL-H-46170,¹³ and MIL-H-83282.¹⁴ This pyrogram (Figure 2) was also used as the standard comparison for the identification of Greentweed No. 964 (Figure 3), Parkers No. N 756 (Figure 4), and Parkers No. N 304 (Figure 5), all of which are commercial nitrile elastomers that are used in the M-140 Gun Mount System, and the M-60 and M-1 tank hydraulic systems. Since field problems consisted of leaks in the gun mount system, the knowledge of whether or not the elastomer was formulated to the standard was required.

It was found that the free seal, T-seal, and quadring were all Greentweed No. 964. The other two types of elastomers in the gun mount were Parker Nos. N-756 and N-304. These two types of elastomers have significant main peaks as shown in Figures 3 and 4. The two hoses were also run, and the 1 $\frac{3}{4}$ -in. diameter was Butyl "N" (Figure 6), and the 1-in. diameter was teflon lined (Figure 7).

The investigation of Greentweed materials and Parker Nos. N 304 and N 756 were necessary in view of research and development involvement in development of the new non-flammable hydraulic fluid. The basic reason is that, since the new non-flammable hydraulic fluid will be used in the same system, these elastomers were checked to see if they would be compatible. The turret and gun control system elastomers used in the M-48 and M-60 series tanks such as the quadrings, Part Nos. 347-815 and 347-815-R-16, and "O" Ring Part No. 347-815-L-03, were all Greentweed No. 964 compound. The seal used with the piston rod wiper, Part No. 347-815-C1M-X1-500033, was Greentweed No. 160. The hydraulic motor seal, Part No. 10923453, was Greentweed No. 964. The two "O" ring packings used in the M-1 tank hydraulic system, Part Nos. M83248/1-908 and M83248/1-916, were both Viton Rubber.

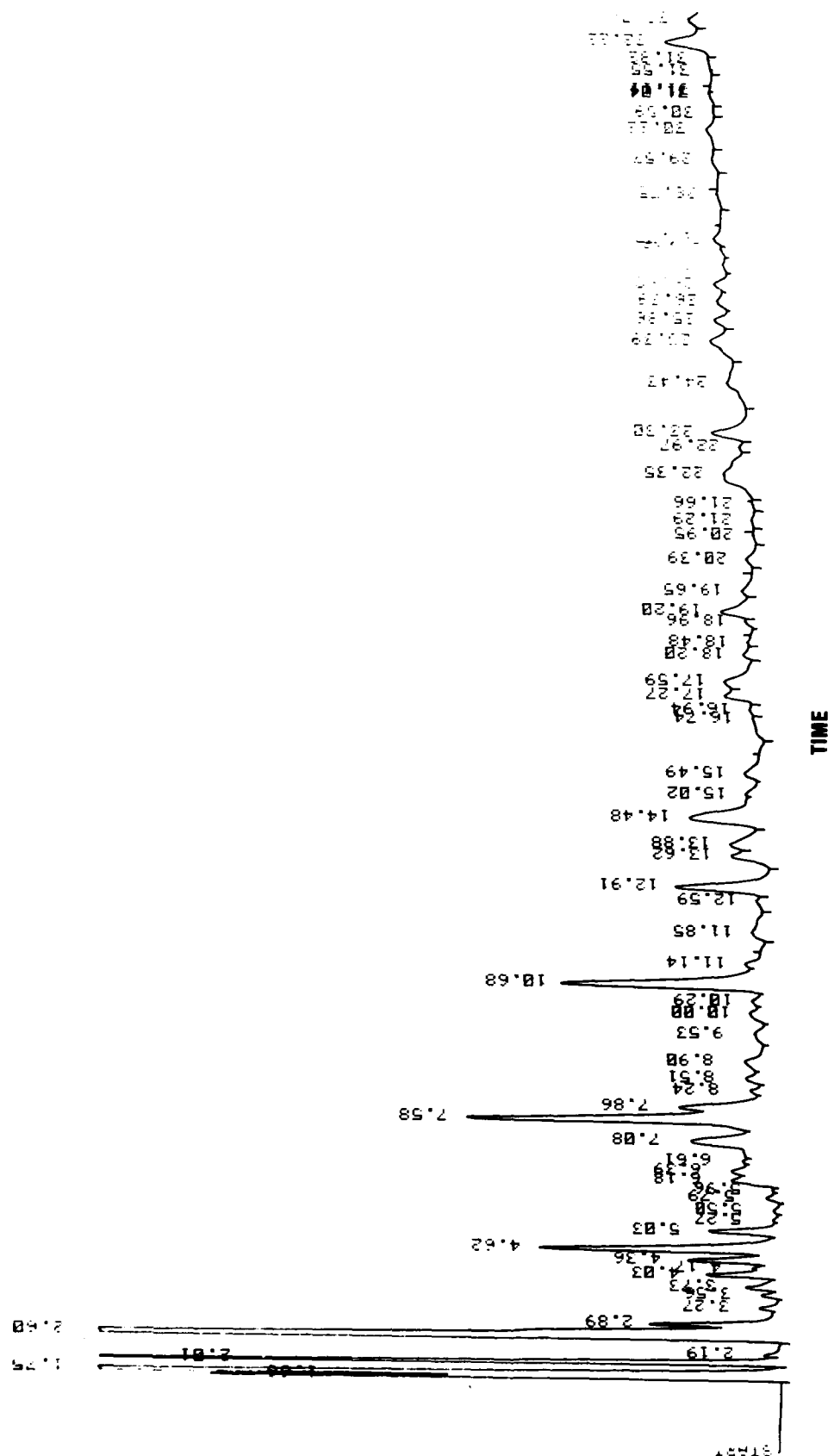
This investigation included Greentweed Nos. GT 160 and GT 964 and Parker Nos. N 304 and N 756 elastomers, since these elastomers are used extensively in the M-60, M-48, and M-1 tank hydraulic systems and gun mounts. Results of this investigation are directly related to the non-flammable Hydraulic Fluid Research and Development program and will be utilized as a tool for identification purposes in the program.

¹¹ Mil-Spec MIL-H-5606, Hydraulic Fluid Petroleum Base; Aircraft Missile, and Ordnance, 29 August 1980.

¹² Mil-Spec MIL-H-6083, Hydraulic Fluid, Petroleum Base, For Preservation and Operation, 28 September 1973.

¹³ Mil-Spec MIL-H-46179, Hydraulic Fluid, Rust Inhibited, Fire-Resistant, Synthetic Hydrocarbon Base, 18 August 1982.

¹⁴ Mil-Spec MIL-H-83282, Hydraulic Fluid Fire Resistant, Synthetic Hydrocarbon Base, Aircraft, 10 February 1982.



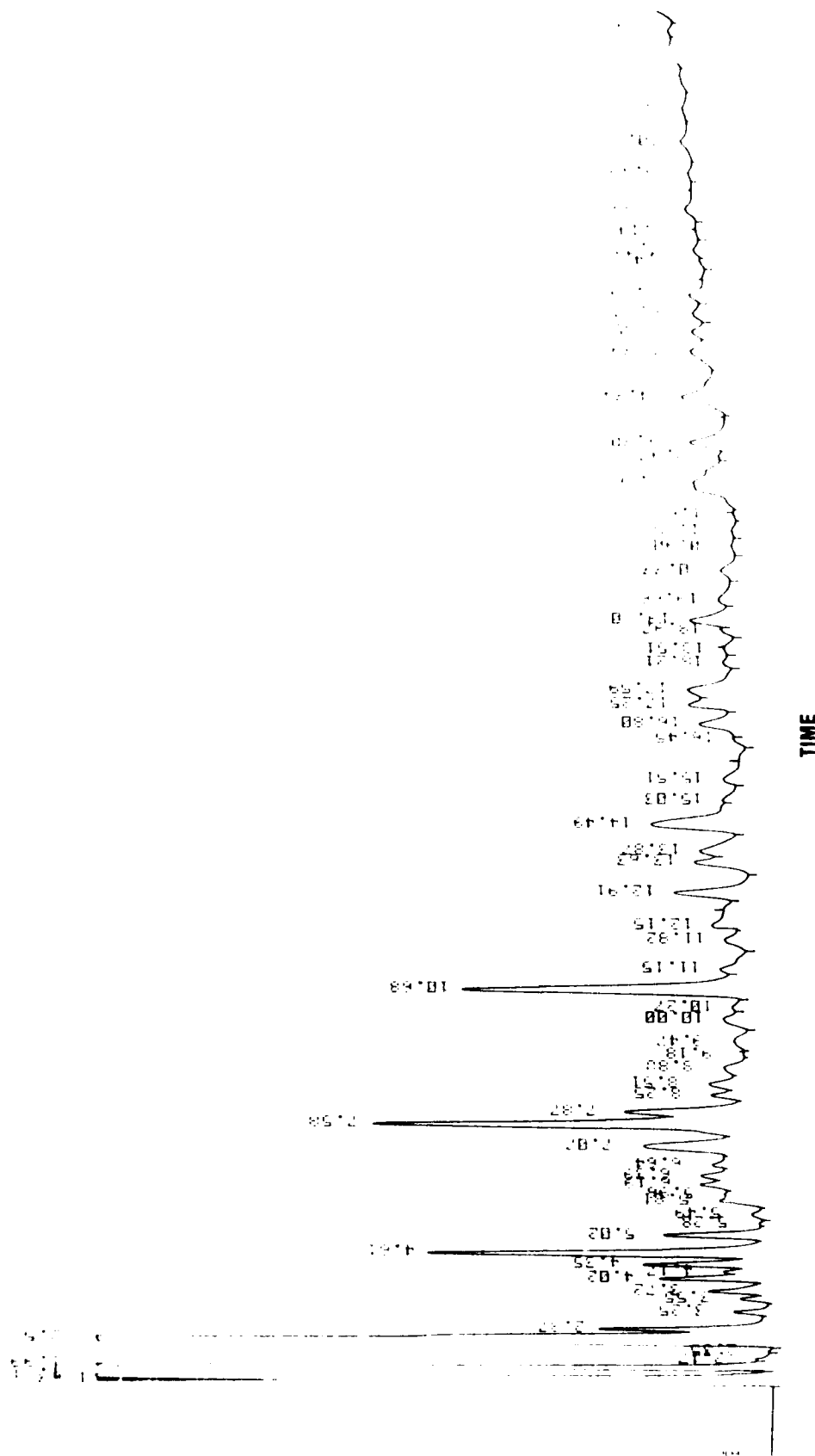


Figure 4. Parkers N-756.

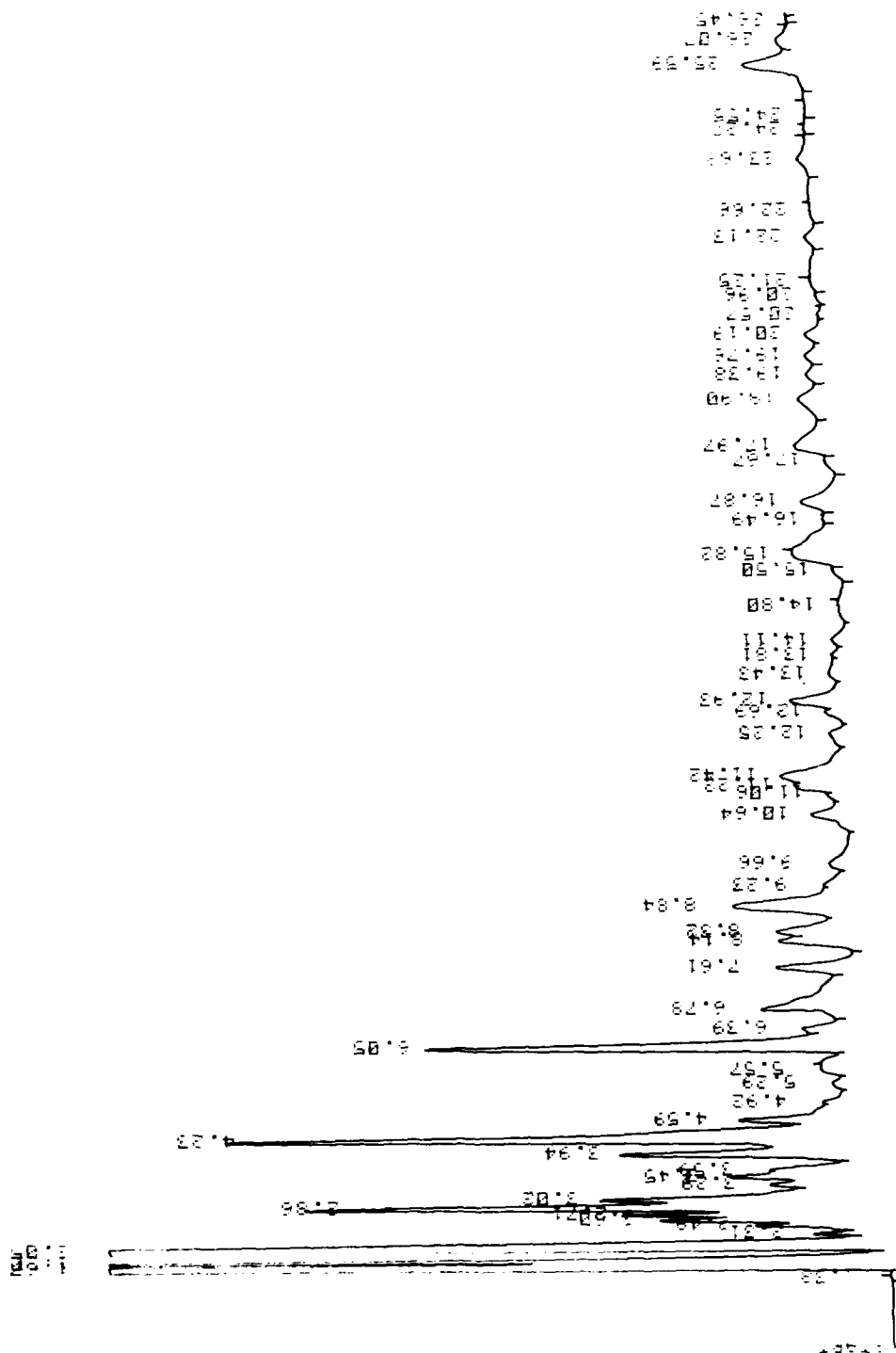


Figure 5. Parkers N-304.

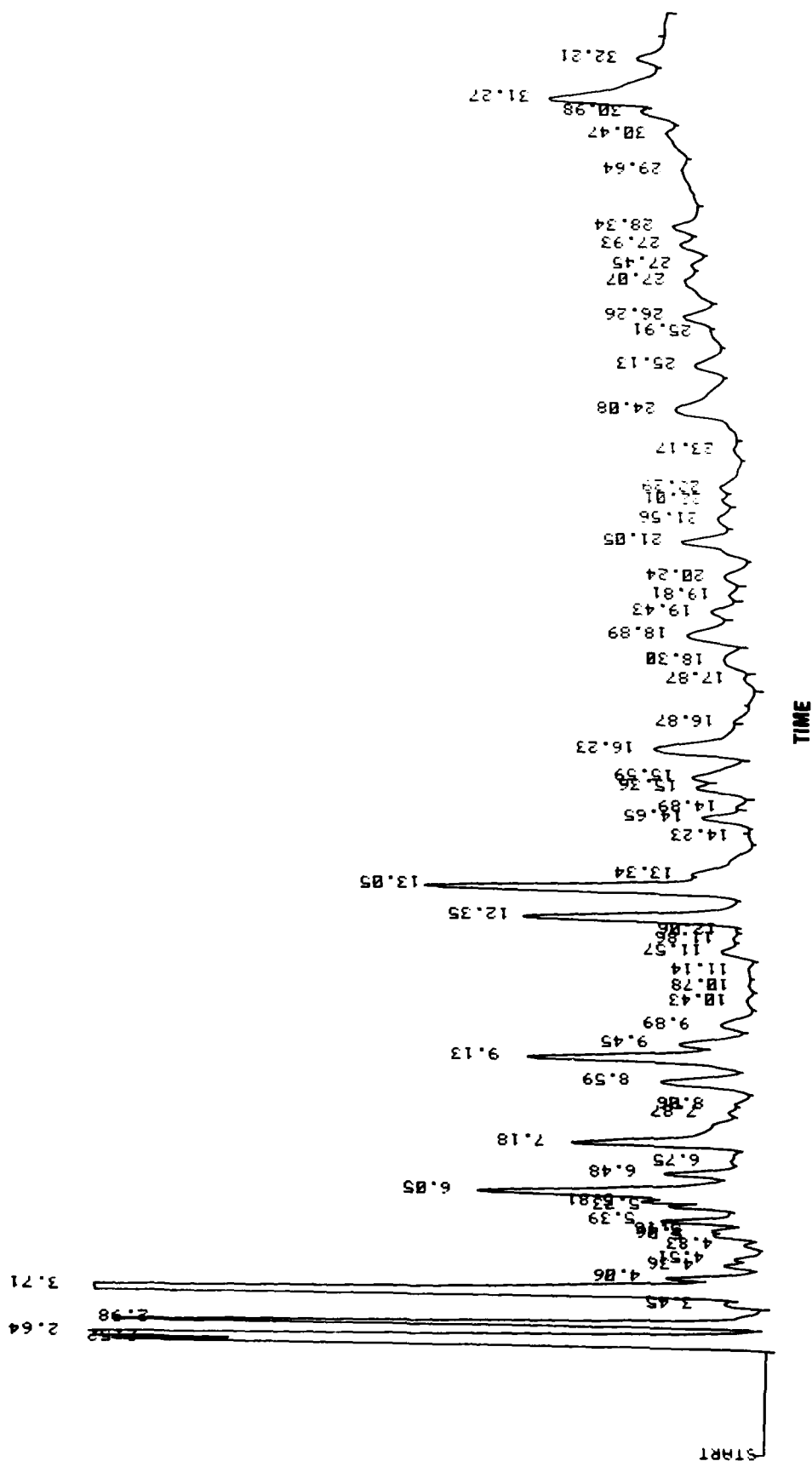


Figure 6. Butyl "N" Rubber.

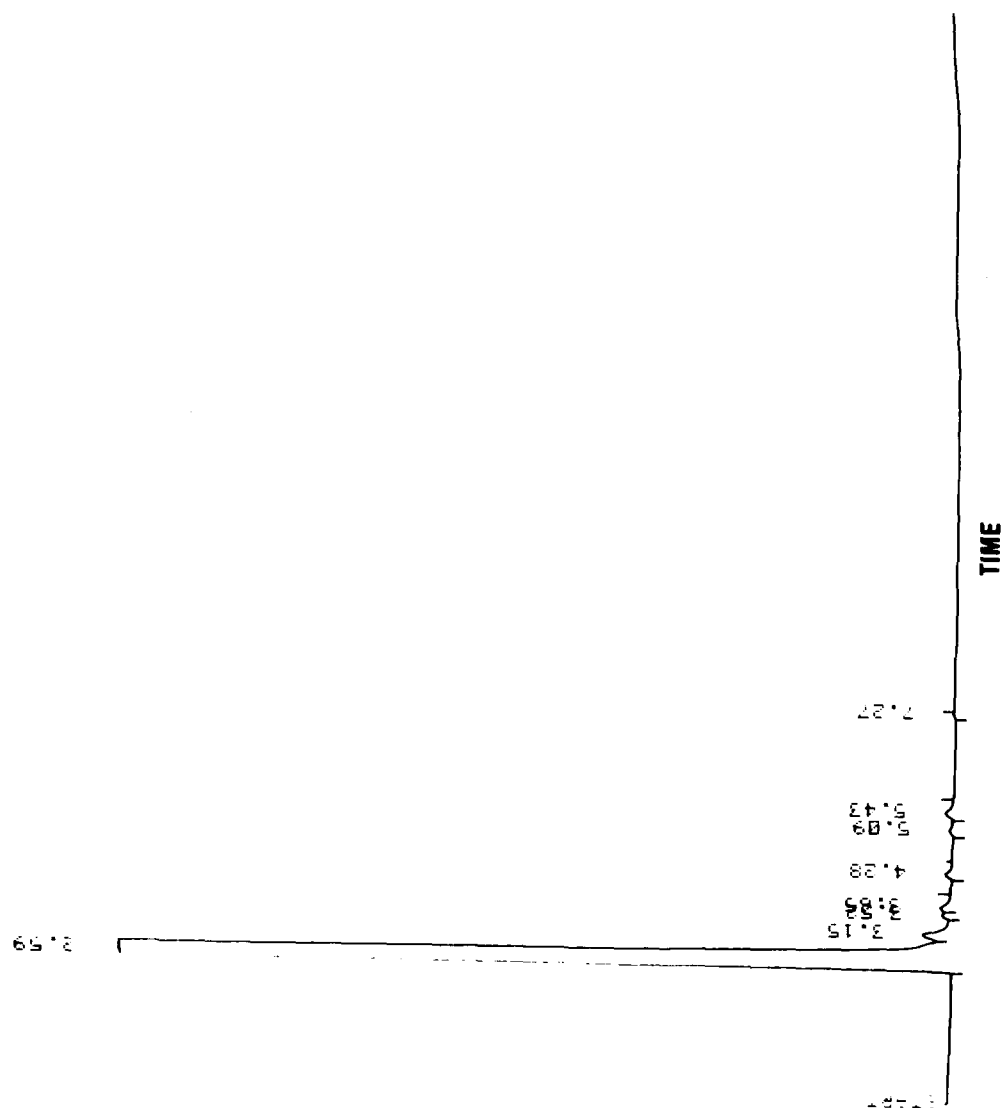


Figure 7. Teflon.

IV. DISCUSSION AND CONCLUSIONS

From the results of this study, careful examination of the various pyrograms shows that it is possible to identify the different elastomers by class. Only a few examples were used to illustrate applications of the proposed procedure. However, the method has broad application to a wide range of elastomeric materials. The automotive industry, out of necessity, utilizes a large variety of different types of elastomers having diverse chemical and physical properties, such as flame, chemical, cold, steam, tear, and abrasion resistance. The specificity of some elastomers for certain application cannot be overstressed, for it is the narrow range of usefulness of many elastomers that imposes the need for rigid quality control. Accordingly, elastomers used in other materials of interest to the military (e.g., gas masks, wearing apparel, fuel tanks, balloons, recoil system of gun mounts, etc.) must conform to definite chemical types, in order to impart the desired performance characteristics to the final product. Pyrolytic gas chromatography has the potential for characterizing the elastomeric composition of most elastomer stocks and synthetic and/or natural rubber in manufactured products such as those described above.

The materials tested contained pigments and fillers that form a residue when the elastomer products were pyrolyzed. Although the pyrolysis reaction was not affected by the quantity and amount of pigment in each sample, an accumulation of residue from repetitive analyses caused polar decomposition products to be adsorbed producing asymmetric peaks on the chromatograms. To maximize the reproducibility and accuracy of the method, the injection port liner should be removed and cleaned after approximately ten analyses.

The procedure described in this report should help expand the testing capability of this laboratory (Fuels & Lubricants) by providing an accurate and rapid method for the characterization of elastomers. It can be used in composition/performance investigations and as a diagnostic tool for trouble-shooting field problems. Other factors that warrant future consideration are:

- (1) The improvement of interlaboratory reproducibility.
- (2) The improvement of quantitative analysis.
- (3) A method for distinguishing between copolymers and physical blends of elastomers.
- (4) A computer.

One approach to the analysis of the data generated by this technique involves the use of a computer to search for representative peaks which can be used to identify particular types of elastomers. This system will, then, automate the data analysis function by selecting unique peaks or groups of peaks for compound identification. This approach will not only substantially decrease the time required for interpretation but will increase the accuracy of this process. Once an elastomer is chromatographed, its pyrogram can be stored for future comparisons, and this will allow routine analysis of elastomers, even from field samples or from samples for which there is little or no other information concerning its origin.

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